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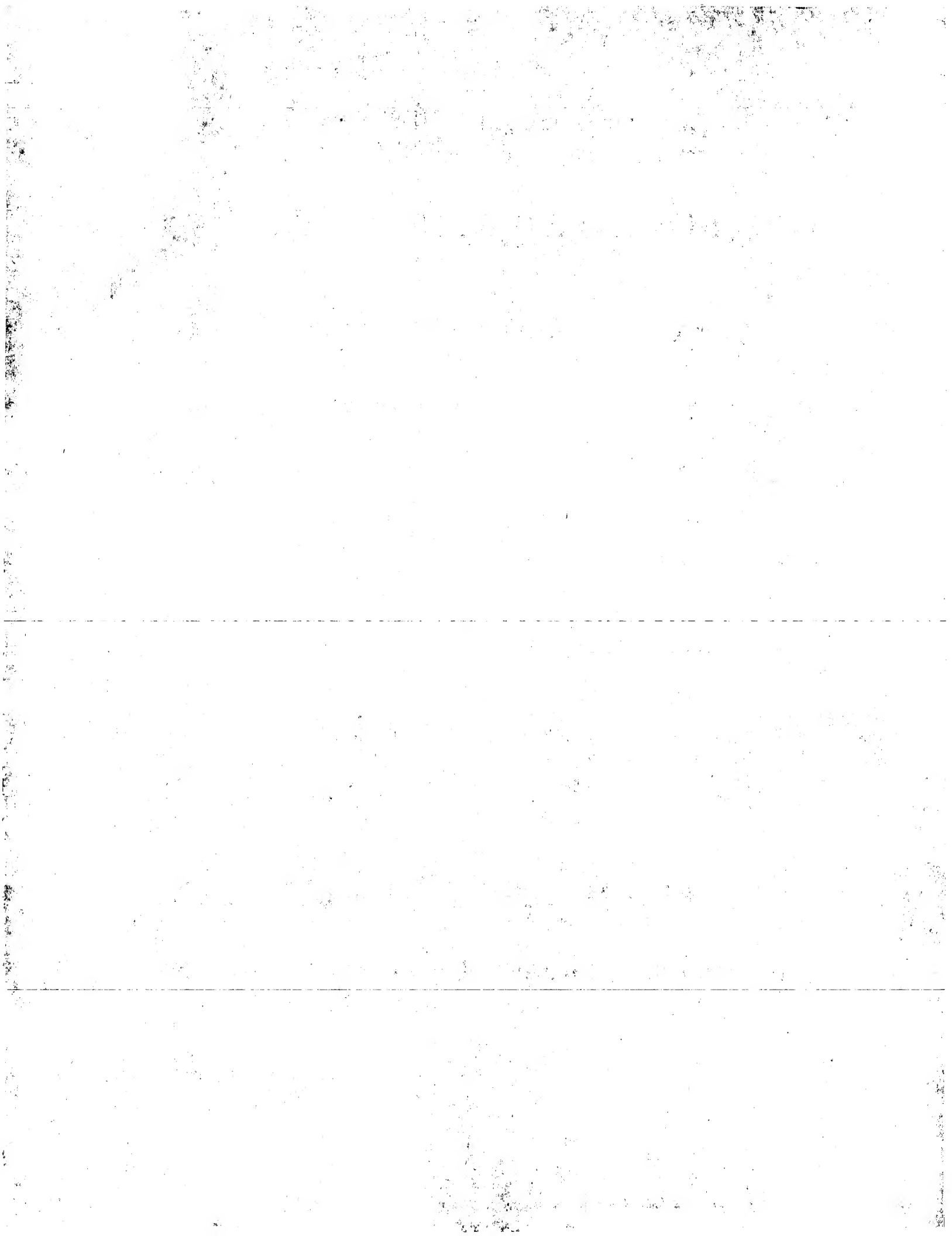
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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(54) Title:</b> ANIONIC DETERGENT PARTICLES  <b>(57) Abstract</b>  High bulk density detergent particles containing high levels of anionic surfactant (e.g. LAS and/or PAS) and a phosphate builder have improved granularity by virtue of also including aluminosilicate builder.		

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ANIONIC DETERGENT PARTICLES

The present invention relates to anionic detergent particles, a process for their production and a composition containing them. More particularly the present invention relates to detergent particles which contain both an anionic surfactant and which contain a phosphate detergency builder.

Detergent-active compounds conventionally employed in detergent compositions include anionic surfactants e.g. linear alkylbenzene sulphonates (LAS), linear alkyl ether sulphate (LES) and primary alkyl sulphates (PAS), and nonionic surfactants e.g. alcohol ethoxylates. To improve detergency performance it is desirable to provide a high level of detergent-active material in the powder, as well as a detergency builder.

Often, the maximum level of active that may be incorporated is limited by process requirements. Detergent compositions having a high bulk density are typically prepared by a process involving mixing or granulation of components of the composition and/or a base powder obtained for example from a spray-drying process and provide significant consumer benefits as compared to compositions of lower bulk density.

It is known to incorporate detergent active compounds into such compositions in liquid form. However as it is necessary to control the ratio of liquids to solids in order to form detergent granules the maximum level of detergent active material which may be incorporated in this manner is limited. It is also known to incorporate anionic surfactant e.g. LAS or PAS in detergent compositions by means of a solid adjunct, that is, a particle comprising the surfactant and other components of the composition e.g. builder. It can be quite difficult to incorporate high levels of anionic

surfactant in such adjuncts whilst maintaining good flow properties and sufficiently low tendency to agglomerate. Several solutions to this problem have been proposed, including that disclosed in our PCT Patent Application No. PCT/EP95/03321 (unpublished before the priority date of the present application).

Although there has been a trend away from use of phosphate builders in favour of other inorganic builders such as aluminosilicates, carbonates or citrates, phosphates may still be desirable in certain formulations on account of their excellent builder properties per unit cost of material. Unfortunately, if relatively large amounts of anionic surfactants such as LAS or PAS are used in conjunction with phosphate builders, the tendency to over-agglomeration again becomes acute, even if production processes designed to avoid over-agglomeration are adopted. This problem is especially marked with LAS. However, high bulk density products usually require high levels of anionic surfactant and above 20% by weight of the anionic, the severity of the problem is unacceptable, without the use of fine particulate material, e.g. zeolite to control the granulation, which allows the anionic level to be to ca 30%..

The aforementioned problem associated with phosphate builders has now been overcome by incorporating aluminosilicate builders in the particles.

US-A-4 524 010 discloses a spray dried detergent granule containing phosphate and aluminosilicate builder units up to 40% by weight of anionic surfactant.

US-A-4 019 999 describes a spray-dried detergent composition with aluminosilicate builder and reduced levels of orthophosphate or pyrophosphate builder. Up to 40%

surfactant may be present, which surfactant may include anionic surfactant material.

5 The present invention provides detergent particles comprising at least 20% by weight of anionic surfactant, together with at least 15% by weight of a phosphate builder, and aluminosilicate builder in and/or on the surface of the particles.

10 As mentioned above, the over-agglomeration problem in the presence of phosphate builder is especially marked when LAS is incorporated. Therefore, in the context of the present invention, the anionic surfactant preferably comprises more than 20% by weight of the total particles of LAS. However,  
15 the anionic surfactant may also consist of or comprise any other anionic surfactant, especially PAS.

The amount of anionic surfactants in the particles is preferably at least 20% by weight, more preferably at least  
20 25% by weight, especially 30% by weight and most preferably, 35% by weight of the particles. In each case, the anionic surfactant may be formed of one or more surfactants selected from LAS, PAS, LES or any other anionic surfactant.

25 The particles preferably contain from 15% to 60% by weight of the phosphate builder, the range of from 20% to 50% by weight being especially preferred.

30 The particles also preferably comprise from 1% to 40% by weight of the aluminosilicate builder, for example 1.5% to 20%.

It is also preferred to incorporate an inorganic co-builder as well as the phosphate and aluminosilicate builders.  
35 Examples of such inorganic co-builders, as well as organic

co-builders are given below. However, it is especially preferred to incorporate into the particles, an inorganic co-builder in the form of sodium carbonate. The inorganic co-builder may for example comprise from 1% to 30% by weight of the particles, more preferably from 2% to 25% by weight.

The phosphate builder may be any inorganic phosphate builder such as hereinafter described as an optional component of a base powder to which the detergent particles according to the present invention are added. However, sodium tripolyphosphate is particularly preferred.

Similarly, the aluminosilicate builder may also be any aluminosilicate, for example any hereinafter described as an optional ingredient of a base powder to which the detergent particles of the present invention are added, either a crystalline or amorphous aluminosilicate. However, zeolites are especially preferred.

Advantageously, a layering agent, e.g. an inert material such as an aluminosilicate may be dosed separately in the cooling zone to coat the particles and thus improve granularity. When the layering agent is an aluminosilicate, this may constitute some or all of the aluminosilicate builder of the particles of the present invention as defined by claim 1. The layering agent can also be any fine particulate material (e.g.  $<25\mu$ ) that will coat the sticky particles to improve their flow properties, e.g. silicas, talc, clays etc.

Detergent compositions of the invention preferably have a bulk density of at least 500 g/l, more preferably at least 550 g/litre, more preferably at least 700 g/litre.

Detergent particles according to the present invention may be prepared either by spray-drying, by post-tower densification of spray-dried powder, or by wholly non-tower methods such as dry mixing and granulation. As used herein, the term "non-tower" refers to any method of mixing and/or granulation which produces particles without spray-drying, although spray-dried materials may be used as starting materials. A high-speed mixer/granulator may advantageously be used for such mixing/granulation. Processes using high-speed mixer/granulators are disclosed, for example, in EP-A-340 013, EP-A-367 339, EP-A-390 251 and EP-A-420 317.

Non-tower production may also be carried out in a flash reactor. Suitable flash reactors include e.g. the Flash Drier system available from VRV SpA Impianti Industriali. Such a reactor preferably has a drying zone which may for example have a heat transfer area of at least 10m<sup>2</sup>. Preferably also, it has a cooling zone which desirably has a heat transfer area of at least 5m<sup>2</sup>. The ratio of drying zone heat transfer area to cooling zone heat transfer area is typically from 3:1 to 1:1, e.g. about 2:1.

The drying zone may optionally be under a slight vacuum to facilitate the removal of water and volatiles. The vacuum may be from 100 Torr up to atmospheric pressure as this provides significant process flexibility. However, a vacuum in excess of 500 Torr up to atmospheric has the advantage of reducing capital investment whilst providing vacuum operation.

Control of residence time and particle size may be secured and process throughput may be increased by agitating the material in the drying and/or cooling zone.

Suitably the average residence time in the drying zone is less than 5 minutes. A residence time of less than 4 minutes is especially preferred with as low a residence time as possible being most preferred.

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The cooling zone is suitably operated at a temperature not in excess of 50°C and preferably not in excess of 40° e.g. 30°C. Desirably there is agitation within the cooling zone to provide efficient cooling of the material therein. By actively cooling the particles, the possibility of thermal decomposition occurring due to the particles being heated to a higher temperature than previously disclosed, is reduced and the tackiness of the particles may be reduced. Such active cooling may for example be effected by circulation of cold water or chilled water (e.g. glycol water at ca. -5°C) around the cooling zone, e.g. in a cooling jacket.

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Optionally two or more drying zones may be employed before the cooling zone as desired. A single apparatus may be employed to provide the drying zone and cooling zone as desired or alternatively separate apparatus for example a drier and a cooling fluid bed may be employed.

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Suitably the drying zone is substantially circular in cross section and is thus defined by a cylindrical wall. Preferably the said wall is heated by means of a heating jacket through which water, steam or oil may be fed. The inside of the said wall is preferably maintained at a temperature of at least 100°C and especially at least 120°C. Preferably the drying zone has an evaporation rate of 2 to 20, and especially 3 to 15 kg water per m<sup>2</sup> of heat surface per hour.

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The cooling zone is preferably defined by a cylindrical wall. Where the process is continuous, the apparatus is

suitably arranged such that the drying zone and cooling zone are substantially horizontally aligned to facilitate efficient drying, cooling and transport of the material through the drying and cooling zones in a generally horizontal direction.

Suitably the drying zone and preferably the cooling zone have agitation means therein which agitates and transports the surfactant paste and forming granules through the said zones. The agitation means preferably comprises a series of radially extending paddles and/or blades mounted on an axially mounted rotatable shaft. Desirably the paddles and/or blades are inclined in order to effect transportation and preferably have a clearance from the inner wall of not more than 10mm, for example 5mm.

LAS is most usually commercially available in the form of the free acid. Unlike PAS acid which is extremely unstable, LAS acid is very stable and sold commercially by a number of suppliers, for example Petralab 550 (Petresa), Deter (Deter), Marlican (Huls), Nalkylene 540L (Vista) and Isorchem L83 (Enichem). It is provided as a viscous liquid that is easily handled, stored and processed.

Neutralised LAS acid is commercially available as a powder.

LAS powders are mainly either drum or spray dried and can have reasonable powder properties when fresh. However they are less preferred because they can absorb moisture from the atmosphere and become sticky and difficult to handle. Their flow deteriorates and they become prone to caking. Typical powders that are available (ex Huls) are Marlan ARL (80% LAS), Marlan A390 (90% LAS), Marlan A396 (96% LAS), or (ex

Unger) Ufaryl DL90 (90% LAS), Ufaryl DL85 (85% LAS), and Ufaryl DL80 (80% LAS).

PAS is presently available on the market in fine powder form or in noodle form. The fine powder is generally dusty, having a significant quantity of particles of less than 150 microns. PAS noodles are generally produced by extruding dried PAS which has the appearance of soap chips and typically have a very large particle size and a very low porosity leading to poor dissolution characteristics. To increase the level of detergent active material in a detergent composition it is known to post-dose detergent particles to provide a composition having a high level of active material.

However, PAS in fine powder form and PAS noodles are generally not suitable for post-dosing into a detergent composition as the composition particles and the post-dosed particles are generally of different particle size and thus tend to segregate and be unsightly. The process according to the present invention enables detergent particles having a high level of detergent active material and suitable porosity and particle size characteristics to be obtained.

Particles according to the present invention preferably have a porosity of from 0% to 20% by volume of the particle and a particle size distribution such that at least 80% of the particles have a D(4,3) particle size of 180 to 1500 microns, preferably 250 to 1200 microns and less than 10% and preferably less than 5% of the particles have a particle diameter less than 180  $\mu\text{m}$ .

Desirably the detergent particles have an aspect ratio not in excess of 2 and more preferably are generally spherical in order to reduce segregation from other particles in a

formulated detergent composition and to enhance the visual appearance of the powder.

Preferably, any LAS anionic surfactant has a chain length of from  $C_8$  to  $C_{16}$ , preferably from  $C_9$  to  $C_{15}$  and most preferably a narrow range of from  $C_{10}$  to  $C_{14}$ .

If present, any PAS surfactant has a chain length of  $C_{10}$  to  $C_{22}$  preferably  $C_{12}$  to  $C_{18}$  and more preferably to a narrow range of  $C_{12}$  to  $C_{14}$ , Coco PAS is particularly desirable.

The detergent particle optionally may further comprise mixtures of the anionic surfactant, phosphate builder and aluminosilicate builder together with other surfactants and/or non surfactant components as desired.

Suitable other surfactants may comprise alkyl benzene sulphonates, linear alkyl ether sulphates, oxo alcohol sulphates for examples  $C_{11}$  to  $C_{15}$  and  $C_{13}$  to  $C_{15}$  alcohol sulphates, secondary alcohol sulphates and sulphonates, unsaturated surfactants for example sodium oleate, oleyl sulphates, olefin sulphonate, or mixtures thereof.

Especially preferred are LAS rich particles, that is particles in which the amount of LAS exceeds the amount of any other surfactant or non-surfactant and more preferably exceeds the total amount of all other surfactant and non-surfactant components.

Generally the sodium salt of the surfactants will be employed. However, it is preferred to form the anionic component by feeding anionic surfactant and neutralising agent to the drying zone or an entry zone immediately before the drying zone and forming the anionic surfactant in-situ.

If the in-situ formation of anionic is performed in the drying zone the heat of neutralisation evolved in the drying zone reduces the requirement for external heating of the drying zone and is advantageous over processes in which surfactant paste is employed as a feedstock.

Moreover, it is advantageous if the precursor acid may be fed to the drying zone in liquid form rather than as an aqueous solution and the neutralising agent may be concentrated. The total amount of water introduced into the drying zone may be reduced significantly as compared to processes in which a surfactant paste is employed. Such pastes may require at least 30% by weight of water in order to be pumpable.

It is also possible to form the detergent particles prior to dosing of the layer agent, directly from a precursor acid feedstock rather than as a two step process involving production of the surfactant and subsequent formation and drying of a paste to form the detergent particles. This is advantageous as the need to produce a surfactant paste, which can present technical difficulties, is avoided as is the need for transport and storage of the paste.

The precursor acid, for example LAS or PAS acid, is suitably fed to the drying zone in the liquid phase. As the precursor acid may be thermally unstable, the neutralisation preferably occurs sufficiently rapidly and substantially completely such that thermal decomposition of the acid due to the elevated temperature is minimised and desirably avoided.

The precursor acid is suitably fed at a temperature of 40 to 60°C to ensure it is in the liquid form but without

encouraging thermal decomposition. The neutralising agent may be fed into the drying zone at any desired temperature.

5 Suitably the neutralising agent is introduced as an aqueous solution or slurry or solid. Conventional neutralising agents may be employed including alkali metal hydroxides for example sodium hydroxide and alkali metal carbonates, for example sodium carbonate, ideally added as a solid material.

10 Using in-situ neutralisation, rapid throughput can be obtained as compared to a process in which a paste containing a pre-neutralised surfactant is employed.

15 Agitation of the precursor and neutralising agent (hereinafter referred to as the feedstocks) in the heating zone generally provides efficient heat transfer and facilitate removal of water. Agitation reduces the contact time between the feedstocks and the wall of the drying zone which, together with efficient heat transfer, reduces the  
20 likelihood of 'hot spots' forming which may lead to thermal decomposition. Moreover, improved drying is secured thus allowing a shorter residence time/increased throughput in the drying zone.

25 Other non-surfactant components which may be present in the detergent particles include dispersion aids, preferably polymeric dispersion aids and more preferably urea, sugars, polyalkyleneoxides; and builders as hereinafter described.

30 If desired the detergent particles may comprise other organic and/or inorganic salts. Suitable materials in salts, preferably sodium, citrates, carbonates, sulphates, chlorides.

It is especially preferred that a salts be present in the particle when the anionic surfactant component comprises LAS.

- 5 The salts may be present at a level of up to 80% and preferably up to 50% by weight of the particles.

10 Particles according to the present invention may also contain, in addition to the detergent-active compound, phosphate and aluminosilicate builders, optionally bleaching components and other active ingredients to enhance performance and properties. Additional post-dosed detergency builder, bleaching components or other ingredients may optionally be post-dosed to the particle.

15 Additional detergent-active compounds (surfactants) which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

20 Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C8-C15; primary and secondary alkyl sulphates, particularly C12-C15 primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulposuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C<sub>8</sub>-C<sub>20</sub> aliphatic alcohols ethoxylated with an average of from 1 to 20 moles ethylene oxide per mole of alcohol, and more especially the C<sub>10</sub>-C<sub>15</sub> primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

The total amount of surfactant present in the detergent composition is suitably from 5 to 60 wt% although amounts outside this range may be employed as desired.

The detergent compositions of the invention generally also contain a detergency builder. The total amount of detergency builder in the compositions is suitably from 15 to 60 wt%, preferably from 12 to 50 wt%.

Inorganic builders may be present as essential components recited in claim 1 or as optional co-builders as appropriate. In any event, these include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate, as disclosed in

GB-A-1 437 950; crystalline and amorphous aluminosilicates, for example zeolites as disclosed in GB-A-1 473 201; amorphous aluminosilicates as disclosed in GB-A-1 473 202; and mixed crystalline/amorphous aluminosilicates as disclosed in GB 1 470 250; and layered silicates as disclosed in EP-B-164 514. The phosphate builders may, for example, be selected from sodium, orthophosphate, pyrophosphate and tripolyphosphate.

Aluminosilicates, whether present as essential ingredients of the particles of the present invention and/or particle

surface layering agents and/or components of a base powder, may suitably be present in a total amount of from 1 to 40 wt% and preferably an amount of from 1.5 to 20 wt%. The zeolite used in most commercial particulate detergent compositions is zeolite A. Advantageously, however, maximum aluminium zeolite P (zeolite MAP) described and claimed in EP-A-384 070 may be used. Zeolite MAP is an alkali metal aluminosilicated of the P type having a silicon to aluminium ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

Organic builders that may be present in the base powder or as co-builders in the particles according to the present invention or post-dosed include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. A copolymer of maleic acid, acrylic acid and vinyl acetate is especially preferred as it is biodegradable and thus environmentally desirable. This list is not intended to be exhaustive.

Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%. The builder is preferably present in alkali metal salt, especially sodium salt, form.

Suitably the builder system comprises a crystalline layered silicate, for example, SKS-6 ex Hoechst, a zeolite, for example, zeolite A and optionally an alkali metal citrate.

Detergent compositions according to the invention may also contain a bleach system, desirably a peroxy bleach compound, for example, an inorganic persalt or organic peroxyacid, capable of yielding hydrogen peroxide in aqueous solution. The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. An especially preferred bleach system comprises a peroxy bleach compound (preferably sodium percarbonate optionally together with a bleach activator), and a transition metal bleach catalyst as described and claimed in EP 458 397A and EP-A-509 787.

The compositions of the invention may contain alkali metal, preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in an amount from 1 to 30 wt%, preferably from 2 to 25 wt%. However, compositions containing little or no sodium carbonate are also within the scope of the invention.

Powder flow may be improved by the incorporation of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer, or sodium silicate which is suitably present in an amount of from 1 to 5 wt%.

The materials that may be present in detergent compositions of the invention include sodium silicate; corrosion inhibitors including silicates; antiredeposition agents such as cellulosic polymers; fluorescers; inorganic salts such as sodium sulphate, lather control agents or lather boosters as appropriate; proteolytic and lipolytic enzymes; dyes; coloured speckles; perfumes; foam controllers; and fabric softening compounds. This list is not intended to be exhaustive.

Detergent compositions of the invention preferably have a bulk density of at least 500 g/l, more preferably at least 550 g/litre, more preferably at least 700 g/litre.

- 5 The invention will now be explained in more detail by way of the following non-limiting Examples.

Processing

A 1.2m<sup>2</sup> VRV machine was used, having three equal jacket sections. Dosing ports for both liquids and powders were situated just prior to the first hot section, with mid-jacket dosing ports available in the final two sections. Zeolite was added via this port in the final section. An electrically-powdered oil heater provided the heating to the first two jacket sections, with oil temperatures between 120°C and 190°C being used. Ambient process water at 15°C was used for cooling the jacket in the final section. Make-up air flow through the reactor was controlled between 10 and 50 m<sup>3</sup>/hr by opening a bypass on the exhaust vapour extraction fan. All experiments were carried out with the motor at full speed, giving a tip speed of about 30m/sec.

A mixture of 2 parts of light ash/3 parts of STP was made-up and dosed via a screw feeder through the single powder dosing port. Two screw feeders were calibrated, one to dose this powder mixture, the other to dose the zeolite for layering. A mono pump was calibrated to dose ambient temperature LAS acid. This was dosed adjacent to the powder mixture, prior to the first hot section.

A Control with no zeolite and twelve examples with zeolite were prepared. Details are given in Table 1. Additional powder properties for some of the Controls and Examples are given in Table 2.

In these Examples, the following abbreviations apply:-

D(4,3)	=	Weighted average mean particle diameter;
BD	=	bulk density;
DFR	=	dynamic flow rate;
% Comp	=	percentage compression;
UCT	=	uncompacted compression test.

Table 1 - Machine conditions and main powder properties of Controls and Examples

	<u>Sample</u>	<u>%LAS</u>	<u>%STP</u>	<u>%CARB</u>	<u>%ZEO</u>	<u>B.D.</u>	<u>D.F.R</u>	<u>%COMP</u>	<u>U.C.T</u>
5	Control	28.9	45.3	25.8	0	1024	68	7.1	0.7
	Ex. 1	38.2	39.8	20.7	1.3				
	Ex. 2	43.1	37.0	17.8	2.1	888	144	3.5	0
10	Ex. 3	49.1	34.0	15.1	1.9	805	148	2.9	0
	Ex. 4	53.4	31.8	13.0	1.8	806	149	2.9	0
	Ex. 5	57.3	30.0	11.1	1.7	771	145	3.5	0.7
	Ex. 6	59.6	27.5	9.1	3.8	771	148	4.1	0
	Ex. 7	62.8	26.0	7.6	3.5	734	138	4.1	0
15	Ex. 8	64.9	24.3	6.2	4.6	707	141	4.7	0
	Ex. 9	66.4	22.8	4.9	5.	703	142	5.3	0
	Ex. 10	68.0	21.7	3.9	6.4	673	141	7.1	0
	Ex. 11	70.0	20.8	3.0	6.1				
	Ex. 12	70.9	19.8	2.2	7.1				

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These results show poor powder properties for the control without zeolite, in particular flow rate. In contrast, the Examples clearly show the benefits of the present invention.

Table 2 - Further powder properties

<u>Sample</u>	<u>D (4,3)</u> <u>microns</u>	<u>&lt;180</u> <u>microns</u>	<u>&gt;1400</u> <u>microns</u>	<u>% AD by</u> <u>analysis</u>	<u>%</u> <u>water</u>
Ex. 2	992	3.5	19.0		
Ex. 3				48.6	1.27
Ex. 4	1258	0.3	35.6		
Ex. 8	1202	0.4	27.5	58.6	1.76
Ex. 9	1056	0	20.6	65.6	2.14
Ex. 12	1072	1.2	22.0	70.4	2.53

In the light of this disclosure, modifications of the described examples as well as other examples, all with the scope of the present invention as defined by the appended claims, will now become apparent to persons skilled in this art.

**CLAIMS:**

1. Detergent particles comprising more than 20% by weight of anionic surfactant, at least 15% by weight of phosphate builder, the particles further comprising aluminosilicate builder.  
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2. Particles according to claim 1, wherein the anionic surfactant comprises more than 20% by weight of the said particles of linear alkylbenzene sulphonate.  
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3. Particles according to either preceding claim, wherein the anionic surfactant comprises primary alkyl sulphate.
4. Particles according to any preceding claim, comprising at least 35% by weight, preferably more than 40% by weight of the anionic surfactant.  
15
5. Particles according to any preceding claim, comprising at least 50% by weight, preferably at least 60%, especially 70% by weight of the anionic surfactant.  
20
6. Particles according to any preceding claim, comprising from 15% to 60% by weight, preferably 20% to 50% by weight of the phosphate builder.  
25
7. Particles according to any preceding claim, comprising from 1% to 40% by weight of the aluminosilicate builder.
8. Particles according to any preceding claim, further comprising inorganic co-builder.  
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9. Particles according to claim 11, where the inorganic co-builder comprises sodium carbonate.  
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10. Particles according to claim 11 or claim 12, wherein the amount of inorganic co-builder is from 1% to 50%, preferably 2% to 35% by weight of said particles.

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11. Particles according to any preceding claim, wherein the phosphate builder comprises sodium tripolyphosphate.

12. Particles according to any preceding claim, produced by a non-tower process.

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# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 97/00590

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C11D17/06 C11D3/06 C11D17/00 C11D3/12

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 351 937 A (UNILEVER PLC ET AL.) 24 January 1990 see page 3, line 39 - line 46 see page 3, line 51 - line 53 see page 5, line 58 see page 6, line 1 see page 6, line 20 - line 28 ---	1-6,8-11
X	EP 0 084 657 A (HENKEL KGAA) 3 August 1983 see the whole document & US 4 524 010 A cited in the application --- -/--	1-11

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents:

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- \*E\* earlier document but published on or after the international filing date
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- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- \*&\* document member of the same patent family

Date of the actual completion of the international search

5 June 1997

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# INTERNATIONAL SEARCH REPORT

Intern. Application No

PCT/EP 97/00590

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	EP 0 353 976 A (PATERSON ZOCHONIS UK LTD) 7 February 1990 see page 4, line 9 - line 15 ---	1-12
Y	DATABASE WPI Section Ch, Week 7813 Derwent Publications Ltd., London, GB; Class D25, AN 78-24250A XP002032333 & JP 53 016 713 A (LION FAT & OIL CO LTD) , 16 February 1978 see abstract ---	1-12
A	WO 95 02036 A (UNILEVER PLC ) 19 January 1995 see claims; examples ---	1-12
P,A	WO 96 38529 A (PROCTER & GAMBLE) 5 December 1996 see the whole document -----	1-12

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